



Consumer and
Corporate Affairs Canada

Consommation
et Corporations Canada

(11) (A) NO. 1 230 825

(45) ISSUED 871229

(52) CLASS 167-10.1
C.R. CL. 4-17.7;
134.40

(51) INT. CL. A01N 43/50

(19) (CA) **CANADIAN PATENT** (12)

(54) Apparatus and Method for Disinfecting Water and
Hydantoin Disinfectant Tablets Therefor

(72) Richards, Randall G.,
U.S.A.

(73) Granted to Bristol-Myers Company
U.S.A.

(21) APPLICATION No. 440,520

(22) FILED 831104

(30) PRIORITY DATE U.S.A. (440,126) 821108

NO. OF CLAIMS 31

Canada

DISTRIBUTED BY THE PATENT OFFICE OTTAWA
CCA-274 (11-82)

1230825

3-1

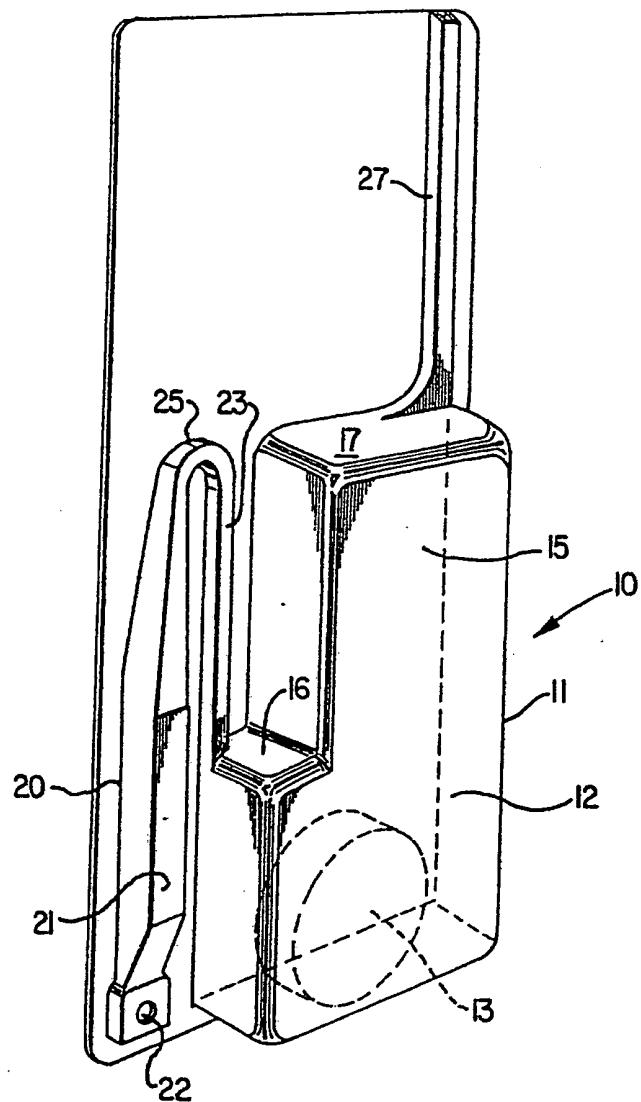


FIG. I

Gowling & Henderson

1230825

3-2

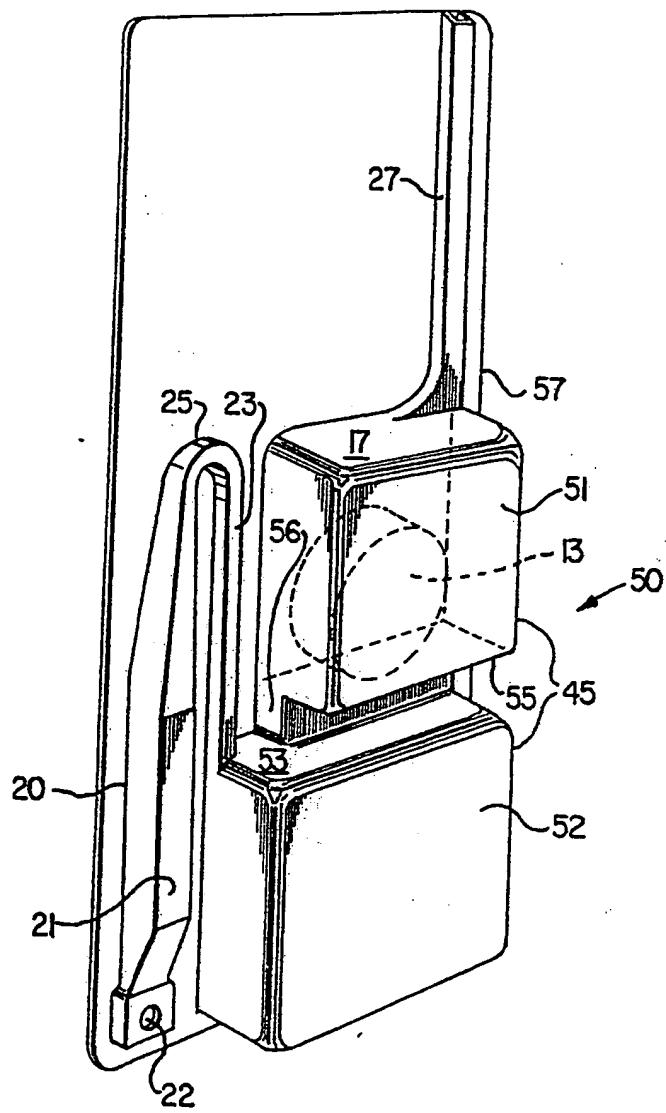


FIG. 2

Gowling & Henderson

1230825

3-3

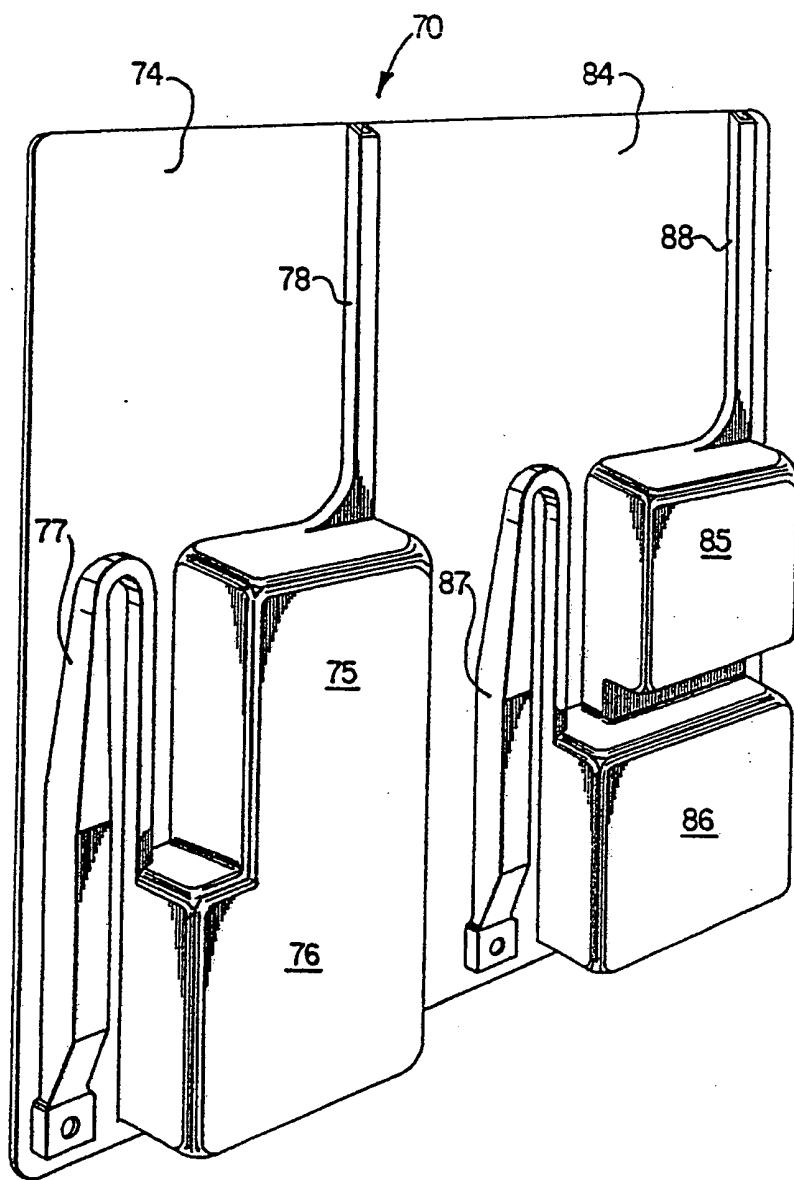


FIG. 3

Gowling & Henderson

FIELD OF THE INVENTION

This invention relates to a tabletted composition comprising an N-halogenated disinfecting agent and a borax binder, and to a method of treating water with said tabletted composition. More specifically, this invention relates to a tablet comprising on a weight basis from about 40 to about 95% of an N-halogenated hydantoin and from about 5 to about 60% borax binder, said tablet having a break strength of at least 35 pounds as determined by a Rimac Spring Tester, and a density of between about 1.5 to 2.0 gm./cm. Most specifically, the invention relates to the tabletted composition defined above suitable for use in a toilet tank dispenser of the type wherein a disinfectant solution is dispensed each time the toilet tank is flushed, and to a method of disinfecting.

BACKGROUND OF INVENTION

The germicidal and disinfecting activity of N-halogenated organic compounds, particularly N-halogenated hydantoin compounds, is well known. U.S. Patent 2,938,764 to Blomfield discloses an improved bleach composition containing dichlorodimethylhydantoin in combination with an alkalinizing agent, e.g., alkali metal hydroxides and silicates. U.S. Patent 3,318,815 to Remler, et al., discloses a toilet bowl cleaning composition having germicidal properties, particular germicides being 1-chloro- and 1,3-dichloro-5,5-dimethylhydantoins. The Remler, et al., composition contains less than 3% of the germicide, and are granulated compositions for pouring into the toilet bowl. Similarly, U.S. Patent 3,338,837 to Hodes discloses an iodinated detergent used in the form of a dilute solution, as a hard surface cleaner.

U.S. Patent 3,147,219 to Paterson discloses N-halogenated organics suitable for use as disinfectants for swimming pools, the disinfectants including hydantoins, provided in tablet form as indicated in Example 1 thereof. However, the tablets contained no binder, and were dissolved rapidly. As pointed out in U.S. Patent 3,412,021, also to Paterson, the compositions of the '219 patent were restricted to single-dosage uses because of the difficulty in metering available halogen. The '021 patent discloses that this metering

1230825

problem may be overcome by using a low-solubility disinfectant, for example, bromochlorodimethylhydantoin, in agglomerate form, the binder being selected from polymers, for example, natural and synthetic rubbers, the polyisobutylenes, the polystyrenes, and the like, and from polyvalent metallic cations, for example, those of aluminum, zinc, tin, iron, chromium, magnesium and silicon.

However, the Paterson agglomerates are of low density and are extremely friable. Hence, because of their porosity, small extrudates, cakes and tablets containing large amounts of disinfectant are not achievable. Furthermore, integrity is poor, resulting in extrudates, tablets and cakes that crack, chip, and break during manufacture and handling. Finally, the Paterson '021 agglomerates, because of the low density and friability, have a dust problem, which requires special consideration during manufacture to reduce both health and safety dangers. Dusting also presents a problem when the agglomerate is to be placed within a two piece plastic container, which container is sealed by heat or radio frequency sealing.

U.S. Patent 4,297,224 to Macchiarolo, et al., discloses a method for the control of biofouling in recirculating water systems, the method utilizing both nonoxidizing and oxidizing biocides. With respect to the hydantoin disinfectant recited in the '224 patent, the agglomerates of Paterson are used. Similarly, U.S. Patent 4,058,618 to Ovchinnikov, et al., discloses the efficacy of dichlorodimethyl and dimethylhydantoins in combination with one another. The Ovchinnikov, et al., composition also comprises an anionic surfactant, a phosphate detergent builder, and sodium chloride and sodium sulfate as diluents to prevent self-heating.

U.S. Patents 4,308,625, 4,249,274, 4,248,827, and 4,200,606, all to Kitko, and 4,216,027 to Wages, each disclose generally various N-organic disinfectants for use in a toilet bowl dispenser. U.S. Patent 4,281,421 discloses a hypochlorite cake in the form of a swellable matrix of limited solubility, the cake comprising the hypochlorite disinfecting agent, and a water-soluble salt of a metasilicate, carbonate and orthosilicate anion, there being at least one cation present that forms an insoluble salt of said anion.

No references are known which use borax as a binder for the N-halogenated compounds of the present invention. However, U.S. Patent 3,883,303 to Roberts discloses a granular composition of borax and paraformaldehyde to treat septic tanks by adding said composition to the toilet bowl. The borax adjusts the pH of the water to optimize paraformaldehyde solubility. U.S. 4,110,431 to Oita discloses a wet weather resistant binder composition comprising a water-soluble polymer and borax, the binder being admixed with a powdered pesticide to promote adherence thereof to foliage. U.S. Patent 3,767,586 to Rutkiewic discloses stable concentrated aqueous solutions of N-halo compounds with borate salts as a weak buffer, while U.S. Patent 2,121,501 to Hershman discloses a borax-containing calcium hypochlorite composition, wherein borax is an optional cleaner constituent. Calcium hypochlorite cakes are disclosed in U.S. Patent 3,793,216 to Dychdala, wherein a hydrated inorganic salt, for example, borax pentahydrate, is incorporated, the concentration thereof being at least 42% by weight, and U.S. Patent 3,406,116 to Vitale disclosed N-chloro hydantoins in a granulated abrasive cleaner composition, borates being optionally includable as an adjuvant.

None of the references cited above, however, considers the problems of providing tablets of N-halogenated hydantoins that do not chip, crack or break, that can be manufactured with a high ratio of active disinfectant to tablet volume, or that minimize the problems of dust generation during manufacture, shipment and handling.

SUMMARY OF INVENTION

The tablets of the present invention comprise on a weight basis from about 40 to about 95% of an N-halogenated hydantoin disinfectant compound, from about 5 to about 60% borax binder, and from about 0 to 10% of a lubricant, the tablets having a break strength of at least 35 pounds on a Rimac Spring Tester. The tablets are substantially dust free and resist breaking, capping and fracturing during normal handling. In water the tablets retain the tabletted configuration for prolonged periods of time, that is for about two to six weeks, when included within a toilet bowl dispenser herein described. Preferably, the tablets comprise between about 20 to 45% by weight borax, and between about 50 to 80% hydantoin. Most preferably, the tablets

comprise 25 to 35% borax and 60 to 75% by weight hydantoin. A lubricant is preferred in a preferred amount of from 0.25 to 5% to facilitate release of the tablet from the tablet press.

The present invention also relates to a dispenser adapted to contain the tablet, the dispenser in general terms comprising a receptacle for the tablet having a water inlet, a solution outlet, and an air vent. Preferred embodiments of the dispenser comprise a solution reservoir having a lower portion and an upper portion, the volume of solution in the upper portion being dispensed by flow through an inlet/discharge conduit adapted to provide siphon or siphon/gravimetric flow of liquid from the reservoir. In the preferred embodiment, the tablet is retained in the upper portion of the reservoir.

In one embodiment the present invention provides such a disinfectant composition comprising on a weight basis:

- (a) from about 40 to 95% of an N-halogenated hydantoin disinfectant selected from the group consisting of 1,3-dichloro-5,5-dimethylhydantoin; 1,3-dibromo-5,5-dimethylhydantoin; 1-bromo-3-chloro-5,5-dimethylhydantoin, and combinations of same,
- (b) from about 5 to about 60% of a borax binder,
- (c) from about 0.25 to about 5.0% of a lubricant, and
- (d) less than about 3% free water moisture,

said tablet having a break strength of from about 35 to about 100 pounds as measured on a Rimac Spring Tester, and a density of from about 1.5 to about 2.0 gm./cc.

In another embodiment the invention provides a method of disinfecting water comprising the steps of providing a disinfectant

1230825

tablet in a container, said tablet comprising on a weight basis from about 40 to 95% of an N-halogenated hydantoin disinfectant; from about 5 to 60% borax binder; less than 10% lubricant, and less than 3% water moisture, said tablet having a break strength of at least 35 pounds on a Rimac Spring Tester and a density of from about 1.5 to about 2.0 gm./cc.; contacting said tablet in said container with water to dissolve a portion of said tablet, and dispensing the resulting solution into the water to be treated.

BRIEF DESCRIPTION OF THE DRAWINGS

Fig. 1 is one embodiment of a dispenser of the present invention containing the tablets of the present invention.

Fig. 2 is a second embodiment of a dispenser of the present invention wherein the tablet is contained in an upper portion of the dispenser solution reservoir.

Fig. 3 is another embodiment wherein two dispensing chambers are provided in tandem, one dispensing chamber containing a surfactant solution, the other chamber containing the disinfectant tablet disclosed herein.

DETAILED DESCRIPTION OF THE INVENTION

The composition of the present invention, which comprises, in tablet form, an N-halogenated hydantoin disinfecting agent and a borax binder, is suitable for use as a water-treating and disinfecting material of general application wherein the tablet is provided in a container, water is brought into contact with the tablet to form a concentrated disinfectant solution, and said solution is released into the body of water to be treated. Hence,

the tabletted composition of the present invention has utility in the treatment of water in swimming pools, industrial process water, ponds, fountains, and the like where a small portion of the water, either as make-up or from the total volume of water present, is circulated continuously through the container and past the tablet of the present invention.

In particular, the tablet can be provided in a container, i.e., a dispenser, for insertion into a small body of water, for example, in a toilet tank, a concentrated solution of the hydantoin disinfecting agent being formed within the container upon filling of the tank after a flush, the concentrated solution being discharged into the tank water, and, ultimately, into the toilet bowl, each time the tank is flushed.

Although agglomerates - i.e., cakes, extrudants and tablets - of N-halogenated hydantoins are known, specifically, those of Paterson, U.S. Patent 3,412,021, prior art products are severely limited in application. The agglomerates of Paterson are quite friable, and result in products of poor integrity. Tablets of Paterson are apt to break, crack or chip during manufacture and handling. This is disadvantageous where one or two tablets are to be provided in a dispenser, e.g., a toilet tank dispenser, in that less than a whole tablet may sometimes be placed in the dispenser, said dispenser then not containing the requisite amount of disinfectant material. Where many tablets are to be placed in a container, i.e., in swimming pool water treating devices, it is undesirable to have tablets of varying size because dissolution will vary in proportion to the surface area exposed. Perhaps more importantly, severe breakage results in a large volume of powder, which is usually discarded as waste.

In addition, the prior art agglomerates tend to disintegrate rapidly, which rapid disintegration is highly undesirable where a single tablet is to last for long periods of time. This problem is particularly acute in toilet tank dispensers where the hydantoin disinfectant tablet should retain its configuration for

two to six weeks, depending on the level of use.

Moreover, the prior art agglomerates are porous, having a very low density. To incorporate a given volume of disinfectant material into a dispenser provided with one or two tablets requires a large container or dispensing chamber, which size is often limited by the tank in which the dispenser is placed. For example, a toilet tank is of limited size and contains various piping and valves. A toilet tank dispenser must be of such size as not to interfere with the proper operation of the valves. Hence, it is necessary to use a tablet of high density providing the requisite amount of disinfecting agent, a problem not addressed by the prior art.

Finally, the tablets should be substantially dust free to minimize problems during manufacture of the tablet and during subsequent use, especially during subsequent incorporation of the tablet into a dispensing container. In particular the problem of dusting is acute where the dispensing container is of two piece plastic construction, the two pieces being joined together by heat or radio frequency sealing. The prior art hydantoin agglomerates are quite dusty in view of their porosity and friability and require suitable precautions during manufacture, shipment and handling.

The tablets of the present invention overcome each of the problems associated with the prior art product. The present invention provides tablets that:

- (a) are substantially dust free;
- (b) have a minimum break strength of 35 pounds on a Rimac Spring Tester, and therefore resist breaking, chipping or fracturing during normal shipment and handling;
- (c) have a density of from 1.5 to 2.0 gm./cc., and hence contain a large amount of the disinfectant

in tablets of small volume notwithstanding
the binder concentration;

- (d) have a high break strength permitting flexibility
as to tablet size and dimensions;
- (e) have dissolution characteristics which can be
regulated by varying the proportions of the
constituents and by regulating density, and
which are

1230825

adapted to retain the tablet configuration in solution over long periods of time;

- (f) resist "capping", that is the tendency of the planar surfaces to shear from the body of the tablet, and "lamination", the tendency of a tablet to break along stria in the body of the tablet, and
- (g) can be fabricated on a commercial tabletting press.

The Tablet

The composition of the present invention comprises on a weight basis from about 40 to about 95% of an N-halogenated hydantoin and from about 5 to about 60% borax binder, said tablet having a break strength of at least 35 pounds as determined on a Rimac Spring Tester, described below in greater detail. Preferably, the tablet further may optionally contain from about 0.25 to about 10% of a lubricant to facilitate release of the tablet from the die. Many materials are known in the art of tabletting which can be used for this purpose, although fatty acids of 10 to 20 carbons and their alkali metal salts are preferred in that they are not readily oxidizable by the hydantoin disinfectant. Preferred lubricants are stearic acid and sodium stearate.

The tablet also contains water associated with the borax, if any, and may also contain free water, that is, water not bound to the borax binder. Various impurities, up to about 10% by weight, for example, alkali and alkaline earth metal halogen salts appearing as by-products in the hydantoin manufacture may also be present. Preferably, the free water is less than about 3% by weight, while the impurities are preferably less than about 5% by weight.

Although borax is the sole binder found which provides tablets of the requisite properties, it should be understood that the impurities mentioned above may have a positive effect on the integrity of the tablet. However, as described below, these impurities as binders do not provide the desired tablet characteristics unless admixed with borax according to the present invention. Finally, other materials used as binders in the tabletting art, while not forming satisfactory

tablets with hydantoin, may not deleteriously affect the tablet of the present invention. It is not intended that the novel benefits derived from the tabletting of hydantoins with borax be restricted by the presence in minor amounts of other constituents. That is, the present invention is to be construed broadly as the tabletting of hydantoins with borax, which provides highly desired properties to the tablet not achieved in its absence.

The hydantoin compounds preferred for incorporation in the tablets of the present invention are 1,3-dichloro-5,5-dimethyl-
10 hydantoin; 1,3-dibromo-5,5-dimethylhydantoin, and 1-bromo-3-chloro-
5,5-dimethylhydantoin. Preferably, the hydantoin is present in the range of from about 50 to about 80% by weight of the tablet, most preferably from about 60 to about 75% by weight. Although the pentahydrate form of borax, $\text{Na}_2\text{B}_4\text{O}_7 \cdot 5\text{H}_2\text{O}$, is preferred, other forms, including anhydrous borax, can be used. Anhydrous borax is hygroscopic and will absorb free water in the composition, or atmospheric moisture, to provide a hydrate. The borax is present preferably in the range of from 20 to about 45% by weight, most preferably from about 25 to about 35% by weight. The borax may be
20 granular or puffed; both being commercial forms of borax. Puffed borax has a density of from 8 to about 50 lbs./ft.³, and is described, for example, in U.S. Patent 3,994,651 to D'Souza. Granular borax, which has a bulk density of about 50 to 70 lbs./ft.³, is preferred.

The tablets can be made with conventional tabletting equipment, for example, Stokes-Penwalt rotary press Model No. DDS-2, by feeding an intimate mixture of the composition described above to the die. Line speeds may be up to 300 tablets per minute, preferably from about 30 to about 200 tablets per minute, with pressures of from about 2
30 tons to about 20 tons, depending upon the percent binder, and desired density and hardness of the tablet. Operation of the tabletting press to obtain the tablets of the present invention may require preliminary trials to establish specific operating conditions for given tablet properties. However, such trials are well within the ordinary skill of one practicing the tabletting art.

In the search for a binder that would provide hydantoin tablets with the desired properties of hardness, density, and integrity, both

when dry and when immersed in water, many substances were considered. Such conventional binders as starch, cellulosic materials, albumin, gelatin, and the like were dismissed because of potential oxidation of the binder by the hydantoin. During manufacture the probability of an exothermic reaction exists with these materials, and their use is considered unsafe. Similarly, other organic compounds with oxidizable sites are considered incompatible with hydantoin, a strong oxidizing agent.

Such incompatibility also exists with many inorganic compounds conventionally used as a binder in the tabletting art, especially at the pressures under which the tablet press operates. Even if the reaction is minimal, the evolution of gases during tabletting presents an undesirable condition. Furthermore, the generation of even small amounts of gas compromises tablet integrity. Finally, the binder selected must be compatible with the hydantoin disinfectant when the tablet is immersed in water.

Hard tablets were made on a Carver Lab press with between about 35 to 39% by weight sodium bisulfate and sodium sulfate at 10 tons pressure, but reacted when immersed in water. Tablets made with 30 to 40% by weight sodium metasilicate on a Carver Lab press disintegrated in water after two minutes. Similar disintegration occurred with tablets containing 30% by weight of sodium carbonate, sodium bicarbonate, and potassium bromide.

Tablets made on a Carver press with 5, 20 and 35% calcium carbonate and 5, 20 and 35% sodium chloride were also unsatisfactory. The calcium carbonate tablets ranged from soft to hard, and were prone to cap and/or flake during handling. Tablets with 35% sodium chloride were hard, but disintegrated in water; 5% NaCl tablets typically were soft, yet did not readily dissolve when immersed; 20% tablets provided tablets that were of moderate hardness and stability. However, all NaCl tablets were prone to cap and/or flake. Furthermore, NaCl at all concentrations tended to leach out in water over time, leaving a hydantoin skeleton that disintegrated. None of the binders referred to above was found to provide a tablet of the desired properties.

Initial trials with borax indicated that hard tablets could be made with a minor degree of capping. It was also found that a tablet

made on a Carver lab press comprising by weight 25% puffed borax (28 lb./ft.³), 10% magnesium nitrate hexahydrate, 64% dibromodimethylhydantoin, and 1% water moisture was relatively stable in water. This tablet was placed in a toilet bowl, which was flushed 10 times over a four-hour period. The borax-containing tablet retained its integrity even under this dynamic testing. Other tablets containing borax and magnesium nitrate were unacceptable, however, in view of poor integrity, and magnesium nitrate was eliminated as a critical constituent.

Tablets containing borax alone were prepared on a Carver lab press at 5, 10, 15, 20 and 35% borax. These tablets had break strengths ranging from about 32 lbs. (Rimac Spring Tester) for a 5% by weight puffed borax (10 lb./ft.³) tablet to over 100 pounds, depending upon the pressure applied in the press and the dwell time in the die to allow air to escape. Other such tablets containing 5% borax had break strengths of from 40 to 55 lbs. Capping for most of these tablets was slight to moderate.

It was also observed that borax tended to be leached from the tablet slowly when immersed, producing a honeycombed tablet but which, unlike sodium chloride, retained its tabletted configuration. This unexpected attribute of borax tabletted hydantoin tablets is preferred, in that a greater surface area of the hydantoin disinfectant is provided after a short period of time in water, and increases the delivery rate of hydantoin, a material of limited solubility, without disintegration of the tablet itself. It is believed that the solubility of the borax is such that removal of this binder does not occur when the tablet is placed in water. Conversely, it is believed that materials having a high solubility, e.g., sodium chloride, are removed from the tablet far more quickly than the depletion of hydantoin. For a binder to be acceptable, it must not only provide the tablet properties desired, but must remain in sufficient quantity to maintain the tabletted configuration over the life of the hydantoin disinfectant. When used in a toilet tank dispenser, the tablet should retain its shape typically for about two weeks to about six weeks, depending on the level of use of the dispenser.

In addition to the materials mentioned above, boric acid tablets were made in a commercial tabletting press. These tablets contained 20 and 25% boric acid. None of the boric acid tablets provided the integrity required, with break strengths substantially below the requisite of 35 pounds on a Rimac Spring Tester.

In addition to the physical attributes of the tablets, an important consideration in the selection of the binder is ease of tabletting in a commercial press. Borax was found to be quite suitable for use in commercial tabletting machines, with a minimum of sticking to the die. Use of a lubricant, however, was found to facilitate removal of the tablets. Sodium stearate and stearic acid are preferred lubricants in that these materials do not have readily oxidizable moieties.

With borax as the binder, the tablets of the present invention resist "capping," that is, the tendency of the planar surfaces to shear from the body of the tablet. Capping occurs because the planar surfaces are not compressed to the same degree as the central core of the tablet during manufacture. We have found that borax is superior to other binders in forming tablets that provide the requisite break strength at the densities stated, and with a minimum of capping. Capping is disadvantageous because the "cap" is likely to form particulate waste that requires disposal, and because the body of the tablet remaining is more easily broken during shipment and handling. Furthermore, capping can decrease the total weight of the tablet by as much as 20%, typically 5 to 15%, which compromises quality control of products incorporating the tablet.

The tablets of the present invention have a break strength, as previously mentioned, of at least 35 pounds, as measured on a Rimac Spring Tester. In this test the tablet is placed on edge between two opposed flat plates and a force applied to the tablet until the tablet fails. The Rimac Spring Tester is manufactured by Rinck-McIlwaine Inc. of Dumont, New Jersey, and is a conventional method of ascertaining break strength. Preferably, the tablets have a break strength of from 50 to about 100 pounds.

Advantageously, the tablets of the present invention have a density of from about 1.5 to about 2.0 gm./cc., preferably from about

1.55 to about 1.8 gm./cc. Notwithstanding high borax concentrations, these tablets have a high ratio of disinfectant to volume, which is desirable when the end product is a container of limited size for immersion in a small body of water. At these densities, the tablets dissolve gradually over time, and are suitable for use in a toilet tank dispenser having a useful life, depending on the level of use, of from 15 to 45 days or longer. Of course, near the end of the life of the tablet, the tablet is weakened by the depletion of materials, and may crumble.

The Dispenser

The toilet bowl dispenser into which the tablet may be placed comprises in most general terms a receptacle containing the tablet, said receptacle having a water inlet, a concentrated disinfectant solution outlet, and an air vent provided in the top of the receptacle.

A preferred embodiment of a dispenser suitable for use in the toilet tank with the tablet of the present invention therein is shown in Fig. 1.

The dispenser 10 of Fig. 1 comprises a solution reservoir 11 having a lower portion 12 for containing the tablet 13 and an upper portion 15 whose volume is equal to the amount of disinfectant solution dispenses. A shoulder 16 is formed at the interface between the lower and upper portions, a siphon inlet/discharge conduit 20 extending upwardly from said shoulder. The conduit 20 comprises an exterior leg 21 that extends below the shoulder 16, said leg 21 having an inlet/outlet port 22 proximate the bottom thereof, and an interior leg 23 extending upwardly from the shoulder 16. The legs 21 and 23 meet in at the arcuate portion 25, preferably at the level of the top 17 of the dispenser 10. The greater the height of the interior leg 23, the less is the amount of diffusion or migration of concentrated disinfectant solution from the solution reservoir 11 during static conditions. The top of conduit 20 may, however, be within the range of the height of the shoulder 16 and the top 17 of the dispenser 10. Vent 27 extends from the top 17 of the dispenser, and is in communication with the atmosphere when the dispenser is in position in the toilet tank.

The dispenser 10 works on the principle of siphon, or on a combination of siphon and gravity discharge, depending upon the height of the conduit 20. Thus, with the conduit 20 at the height of the top 17, all of the solution in the upper portion 15 is dispensed by siphon flow. When the level of the solution reaches the shoulder 16, and air entering through vent 27 enters conduit 20, flow from the dispenser 10 terminates. Conversely, with the top of the conduit 20 at a point between the top 17 and the shoulder 16, that portion of the solution above the top of the conduit 20 discharges by gravity flow, while the portion below the top of the conduit discharges by siphon flow.

Fig. 2 illustrates a most preferred embodiment of the dispenser of the present invention. In this embodiment the solution reservoir 45 of the dispenser 50 comprises an upper portion 51 and a lower portion 52, the shoulder 53 being formed at their intersection. The conduit 20 extends from the shoulder as described above with respect to the embodiment of Fig. 1. Similarly, the vent 27 extends from the top 17 of the dispenser 50.

In this embodiment a shelf 55 is provided between the upper and lower portions of the solution reservoir, the shelf projecting partially into the dispenser such that an elongate pathway 56 is formed between the shelf and the back wall 57 of the dispenser 50. The disinfectant tablet 13 is provided in the upper portion 51.

This location of the disinfectant cake is preferred because a more concentrated solution is dispensed. It is believed that a concentration gradient exists within the dispenser, the disinfectant solute having a tendency to gradually settle towards the bottom. With the tablet in the lower section, the solution dispensed is the solute lean liquid in the upper portion as admixed during dispensation with the solute rich liquid in the lower portion. With the tablet in the upper portion, the solute rich liquid is in the upper portion of the chamber.

Fig. 3 illustrates a dispenser 70 having a first section 74 comprising the dispenser 10 of Fig. 1 and a second section 84 comprising the dispenser 50 of Fig. 1, said dispenser 70 being fabricated from a first sheet of plastic thermoformed to provide the upper and lower portions 75, 76, 85 and 86, the conduits 77 and 87,

and the vents 78 and 88, and a second backing sheet of plastic (not shown) affixed to the first sheet by radio frequency sealing. In this embodiment, the tablet of the present invention is provided in upper portion 85 of section 84, while a detergent cake, known in the art, is provided in the lower portion 76 of section 74. When placed in the toilet tank, both disinfectant and detergent solution are released to the bowl water. Known surfactant cakes suitable for use in the dual chamber dispenser of the present invention are disclosed in U.S. Patent 4,216,027 to Wages.

- 10 The dispenser of the present invention is adapted to deliver preferably between 10 and 100 ml. of concentrated solution of disinfectant such that the concentration of disinfectant in the bowl after the flush is between 2 and 15 ppm., for between 15 to 45 days or longer based on an average use of 10 to 20 flushes per day. To achieve this level of disinfectant concentration in the bowl water, the tablet should contain between about 10 to 50 grams of the disinfectant. Conveniently, the tablet is between 3/4 to 1-3/4 inch in diameter and from 3/8 to 1-1/4 inches high. However, these dimensions are not critical. The dispenser can easily be fabricated
- 20 in plastic by thermoforming to suitable dimensions that fit within most toilet tanks.

EXAMPLE I

Tablets were made on a Stokes-Penwalt DS-3 fourteen station ten ton rotary tabletting press using a 1 inch diameter straight walled die. The tablets were made with bromochlorodimethylhydantoin and either 20 lb./cu. ft. or 40 lb./cu. ft. puffed borax. The tablets had a moisture content of from about 1 to about 3% by weight. Three tablets from each run were weighted, measured for size, and tested for break strength after aging for at least 24 hours. The averages of the

30 readings are provided below in Table I. In run no. 8, the tablet also contained 3% calcium chloride.

1230825

Table I

<u>Run No.</u>	<u>Borax Density (lb./ft.³)</u>	<u>Borax (wt. %)</u>	<u>Average Tablet Weight (gm.)</u>	<u>Average Tablet Thickness (cm.)</u>	<u>Average Break Strength (lbs.)</u>
1	20	10	8.53	1.07	65
2	20	20	7.68	1.17	23
3	40	10	9.18	1.13	57
4	40	10	9.10	1.15	53
5	40	10	9.43	1.20	55
6	40	20	9.22	1.10	71
7	40	30	10.73	1.20	76
8	40	10	9.76	1.15	55

With respect to run no. 2, the low break strength is attributable to the low density of the tablet, calculated as 1.3 g./cc. Each of the other runs, nos. 1 and 3 to 8 provided tablets with densities ranging from 1.55 to 1.76 g./cc.

Overall tablet integrity (dry) was varied from very slight capping (run no. 6) to moderate capping (run nos. 1 and 8). The latter two runs had a relatively low ratio of binder to disinfectant.

EXAMPLE II

Tablets were made with bromochlorodimethylhydantoin on a Stokes-Penwalt single station press using a 1 inch tapered die, and are reported in Table II. The feed mixture contained 68% hydantoin, 31% granular borax and 1% sodium stearate.

1230825

Table II

<u>Run No.</u>	<u>Force (tons)</u>	<u>Tablets/minute</u>	<u>Wt. (gm.)</u>	<u>Thickness (in.)</u>	<u>Break Strength (lbs.)</u>	<u>Tablet Density (g./cc.)</u>
1	<2	15	10.5	0.532	10	1.53
2	2	15	10.6	0.515	30	1.60
3	4	15	10.3	0.483	45	1.66
4	6	15	10.6	0.480	95	1.71
5	6	20	10.5	0.475	70	1.71
6	6	30	10.6	0.485	42	1.69
7	8	-	10.1	0.455	80	1.72

Tablet integrity was good, with slight capping. The tablets had shiny sides, and did not stick to the die or punch. The tablets of run no. 6 had slight lamination marks. With respect to run no. 1, it is believed that the force applied was insufficient to obtain tablets of high break strength.

Tablets were made as above with 78% bromochlorohydantoin, 21% borax and 1% sodium stearate:

Table III

<u>Run No.</u>	<u>Force (tons)</u>	<u>Tablets/minute</u>	<u>Wt. (gm.)</u>	<u>Thickness (in.)</u>	<u>Break Strength (lbs.)</u>	<u>Tablet Density (g./cc.)</u>
8	6	-	10.2	0.474	50-75	1.66

Tablets were made as above with 73% bromochlorohydantoin, 26% granular borax and 1% sodium stearate:

1230825

Table IV

<u>Run No.</u>	<u>Force (tons)</u>	<u>Tablets/minute</u>	<u>Wt. (gm.)</u>	<u>Thickness (in.)</u>	<u>Break Strength (lbs.)</u>	<u>Tablet Density (g./cc.)</u>
9	6	-	10.2	0.470	75-80	1.68
10	6	-	10.3	0.473	75-80	1.69
11	6	-	10.0	0.462	42-55	1.67
12	6	-	10.0	0.470	90-95	1.65

The tablets of runs 8 to 12 had shiny sides, with little capping or sticking.

The tablets made below contained 68% bromochlorohydantoin, 31% granular borax and 1% sodium stearate.

Table V

<u>Run No.</u>	<u>Force (tons)</u>	<u>Tablets/minute</u>	<u>Wt. (gm.)</u>	<u>Thickness (in.)</u>	<u>Break Strength (lbs.)</u>	<u>Tablet Density (g./cc.)</u>
13	6	15	11.55	0.535	75-85	1.67
15	6	15	11.55	0.538	50-55	1.66
16	6	20	11.65	0.545	50-55	1.66
17	7	20	11.8	0.545	72-85	1.68
18	8	20	11.5	0.532	110	1.67

Tablets of runs 13-18 showed some capping, but were generally satisfactory.

EXAMPLE III

Two 10 gram tablets of the present invention were sealed within a dispenser of the type shown in Fig. 1, and two 10 gram tablets were sealed in a dispenser of the type shown in Fig. 2. All four tablets comprised 68% bromochlorodimethylhydantoin, 31% borax, and 1% sodium stearate, and were 1 inch diameter and approximately 1/2 inch thick.

1230825

The dispensers were suspended in separated toilet tanks, which were flushed every hour for 16 hours over a 16 day period. Readings of available halogen level in the bowl water were recorded daily as shown in Table VI.

Table VI

<u>Day</u>	<u>Dispenser of Figure 1</u>	<u>Halogen (ppm)</u> <u>Dispenser of Figure 2</u>
1	5.1	8.3
2	5.5	5.4
3	5.7	5.4
4	3.2	5.7
5	3.5	4.6
6	4.0	4.9
7	4.0	5.9
8	3.6	4.6
9	3.5	4.7
10	3.1	4.6
11	2.0	4.6
12	2.7	4.6
13	2.5	4.3
14	3.5	4.4
15	3.5	-
16	<u>3.5</u>	<u>4.4</u>
Average	3.7	5.1

The preferred embodiment of Fig. 2 delivered about 38% more halogen than the embodiment of Fig. 1. Both dispensers, however, delivered an effective amount of disinfectant to the bowl water.

1. A tabletted disinfectant composition comprising on a weight basis:
 - (a) from about 40 to about 95% of an N-halogenated hydantoin disinfectant, and
 - (b) from about 5 to about 60% of a borax binder,
said tablet having a break strength of at least 35 pounds as measured on a Rimac Spring Tester.
2. The composition of Claim 1 wherein the hydantoin is present in an amount of from about 50 to about 80% by weight and wherein the borax is present in an amount of from about 20 to about 45% by weight.
3. The composition of Claim 2 further comprising a lubricant selected from the group consisting of carboxylic acids of from 10 to 20 carbons and alkali metal carboxylic acid salts of from 10 to 20 carbons, the lubricant being present in an amount of from about 0.25 to about 10%.
4. The composition of Claim 3 wherein the lubricant is selected from the group consisting of stearic acid and sodium stearate.
5. The composition of Claim 1 wherein the tablet density is from about 1.5 to about 2.0 gm./cc.
6. The composition of Claim 1 wherein the N-halogenated hydantoin is selected from the group consisting of 1,3-dichloro-5,5-dimethylhydantoin; 1,3-dibromo-5,5-dimethylhydantoin; 1-bromo-3-chloro-5,5-dimethylhydantoin, and mixtures thereof.
7. The composition of Claim 6 wherein the N-halogenated hydantoin is 1-bromo-3-chloro-5,5-dimethylhydantoin.
8. The composition of Claim 7 wherein the borax binder is puffed or granular borax.

9. The composition of Claim 8 wherein the tablet density is from about 1.55 to about 1.8 gm./cc.
10. The composition of Claim 9 wherein the borax is sodium borate pentahydrate in granular form.
11. The composition of Claim 6 wherein impurities are less than about 10% of the composition.
12. The composition of Claim 11 wherein the impurities are less than about 5% of the composition.
13. A tabletted disinfectant composition comprising on a weight basis:
 - (a) from about 40 to 95% of an N-halogenated hydantoin disinfectant selected from the group consisting of 1,3-dichloro-5,5-dimethylhydantoin; 1,3-dibromo-5,5-dimethylhydantoin; 1-bromo-3-chloro-5,5-dimethylhydantoin, and combinations of same,
 - (b) from about 5 to about 60% of a borax binder,
 - (c) from about 0.25 to about 5.0% of a lubricant, and
 - (d) less than about 3% free water moisture,said tablet having a break strength of from about 35 to about 100 pounds as measured on a Rimac Spring Tester, and a density of from about 1.5 to about 2.0 gm./cc.
14. The composition of Claim 13 wherein the hydantoin is present in an amount of from about 50 to about 80% by weight, and wherein the borax is present in an amount of from about 20 to about 45% by weight.
15. The composition of Claim 14 wherein the tablet has a density of from about 1.55 to about 1.8 gm./cc.
16. The composition of Claim 15 wherein the lubricant is selected from the group consisting of carboxylic acids and alkali metal

salts of carboxylic acids, having from 10 to 20 carbon atoms, the lubricant being present in an amount of from about 0.5 to about 3.0% by weight.

17. The composition of Claim 15 wherein the borax is selected from the group consisting of anhydrous sodium borate; sodium borate pentahydrate; sodium borate decahydrate, and mixtures thereof.
18. The composition of Claim 17 wherein impurities are less than about 10% of the composition by weight, and wherein water moisture is less than 1% by weight.
19. The composition of Claim 18 wherein the borax is sodium borate pentahydrate.
20. The composition of Claim 19 wherein the hydantoin is 1-bromo-3-chloro-5,5-dimethylhydantoin.
21. The composition of Claim 20 wherein the hydantoin is between about 60 and 75% by weight of the tablet, and wherein the borax is between about 25 and 35% by weight of the tablet.
22. The composition of Claim 15 wherein the tablet is between about 3/4 to about 1-3/4 inch in diameter and from about 3/8 to 1-1/4 inch in height, said tablet containing between 10 to about 50 grams of the hydantoin disinfectant.
23. A method of disinfecting water comprising the steps of providing a disinfectant tablet in a container, said tablet comprising on a weight basis from about 40 to 95% of an N-halogenated hydantoin disinfectant; from about 5 to 60% borax binder; less than 10% lubricant, and less than 3% water moisture, said tablet having a break strength of at least 35 pounds on a Rimac Spring Tester and a density of from about 1.5 to about 2.0 gm./cc.; contacting said tablet in said container with water to dissolve a portion of said tablet, and dispensing the resulting solution into the water to be treated.
24. The method of Claim 23 wherein the container is placed in a toilet tank.

1230825

25. The method of Claim 24 wherein the container is filled following a flush and solution is dispensed upon flushing, the toilet bowl water having an available halogen concentration of from about 2 to about 15 ppm. immediately following the flush.
26. The method of Claim 25 wherein the tablet is between about 3/4 to 1-3/4 inches in diameter and between 3/8 to 1-1/4 inches in height, and contains between 10 and 50 gms. hydantoin, and wherein between about 10 to 100 ml. of disinfectant solution is discharged from the container.
27. The method of Claim 26 wherein the tablet has a density of from about 1.55 to about 1.8 gm./cc., and the tablet break strength is from about 50 to about 100 pounds.
28. The method of Claim 27 wherein the hydantoin is present in an amount of from about 60 to about 75% by weight, and the borax is present in an amount of from about 25 to about 35% by weight, the borax being sodium borate pentahydrate.
29. The method of Claim 28 wherein the disinfectant is 1-bromo-3-chloro-5,5-dimethyl hydantoin.
30. The method of Claim 26 wherein the tablet has a useful life of between about 15 to about 45 days of normal use.
31. The method of Claim 25 further comprising the step of dispensing from a second reservoir a surfactant solution.

5
✓

ABSTRACT

This invention relates to a tabletted composition comprising an N-halogenated disinfecting agent and a borax binder, and to a method of treating water with said tabletted composition. More specifically, this invention relates to a tablet comprising on a weight basis from about 40 to about 95% of an N-halogenated hydantoin and from about 5 to about 60% borax binder, said tablet having a break strength of at least 35 pounds as determined by a Rimac Spring Tester, and a density of between about 1.5 to 2.0 gm./cm. Most specifically, the invention relates to the tabletted composition defined above suitable for use in a toilet tank dispenser of the type wherein a disinfectant solution is dispensed each time the toilet tank is flushed, and to a method of disinfecting.

THIS PAGE BLANK (USPTO)